भारतीय मानक
Indian Standard

IS 11678 : 2022

वस्त्र रंजक सामग्री — रंजित वस्त्रादि में नील की तीव्रता के निर्धारण की विधि

(पहला पुनरीक्षण)

Textiles — Method for Determination of Strength of Indigo on Dyed Textiles

(First Revision)

ICS 59.040; 71.040.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली –110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

September 2022 **Price Group 3**

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

Indigo, on account of its fastness to light and washing, beauty of shade and other special propertiesis predominantly used in colouring of textiles. In view of this and of its high price, the accurate estimation of the amount of pure colouring matter present in commercial indigo is very important. The prevalent practice of 'topping' or 'bottoming' indigo dyed materials with other colouring matters has called forth necessity for methods for ascertaining the quantity of indigo present on such dyed materials.

The methods prescribed in this standard are based on the extraction of indigo from the dyed textile material by use of pyridine or cresol mixture at the boiling point of the solvent. The accuracy ofthe method is not affected due to the presence of other dyestuffs in addition to indigo.

This standard was first published in 1986. The first revision has been made in the light of experience gained since its publication and to incorporate the following major changes:

a) Title of the standard has been modified;
b) Grade and purity of chemicals used have been specified; and
c) References to Indian Standard have been updated.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard
TEXTILE DYESTUFFS — METHOD FOR DETERMINATION OF STRENGTH OF INDIGO ON DYED TEXTILES
(First Revision)

1 SCOPE

This standard prescribes a method for determining percentage strength of indigo on dyed wool, cotton and linen textile, when present alone or with other dyestuffs.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No. *Title*
1070 : 1992 Reagent grade water – Specification (*third revision*)
11636 : 2021 Textile dyestuffs — Methods for determination of strength of indigo in substance (*first revision*)

3 PRINCIPLE

3.1 A suitable quantity of the textile material is extracted with pyridine or cresol mixture at its boiling point. The precipitate obtained on filter is washed successively with hot 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution (1 percent), hot water, alcohol, and finally alcohol and ether. The precipitate is then dried and weighed. An alternative method is to render the precipitate of indigo obtained soluble in water by sulphonation with pure concentrated sulphuric acid and titrating with N/50 potassium permanganate solution and then calculating the amount of indigo by the factor: 1 ml of N/50 $KMnO_4$ is equal to 0.00147 g of indigotin.

4 PREPARATION OF TEST SPECIMEN

4.1 The mass of the specimen of the cloth or yarn or fibre to be taken shall be such that it gives from 0.03 to 0.10 g of indigo after extraction. This will vary from 3 to 15 g depending upon the depth of indigo present on the textile material. The material shall be loosely packed into the inner tube of the Soxhlet apparatus (*see* **5.1**). If the material to be tested is cloth, it shall be cut into small strips or pieces and then packed into the inner tube in the form of roll.

5 APPARATUS

5.1 Soxhlet Apparatus — As shown in Fig. 1 which consists of air condenser, in which is placed the inner tube containing the textile material. The inner tube may have the various forms (A, B or C) as shown in Fig. 2. In the apparatus of form A, the hot extract is syphoned intermittently into the distilling flask. With form B, the solvent is directed to the bottom of the tube by means of a long funnel, and then rises through the material overflowing through the side opening near the top. In the form C, it percolates continuously through the materia1, flowing out by the small hole at the bottom of the tube. The simplest and preferable form is tube C as it gives good results with all cloths and is easy to make. The tube is drawn out at the end, and some glass points are fused on to the outer surface so as to

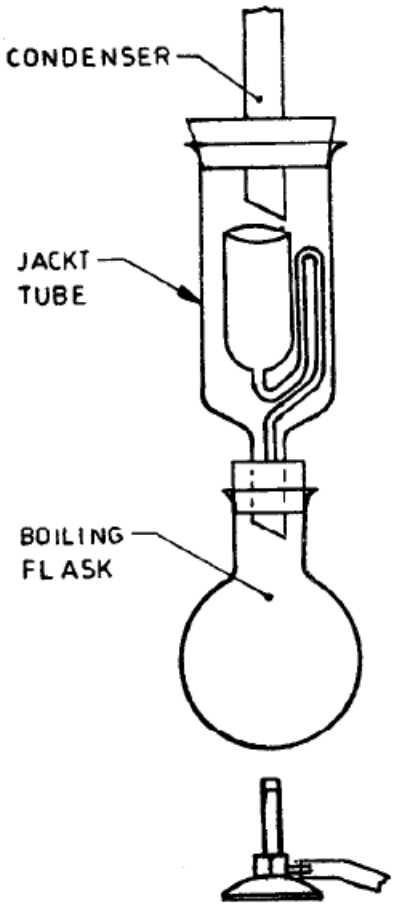
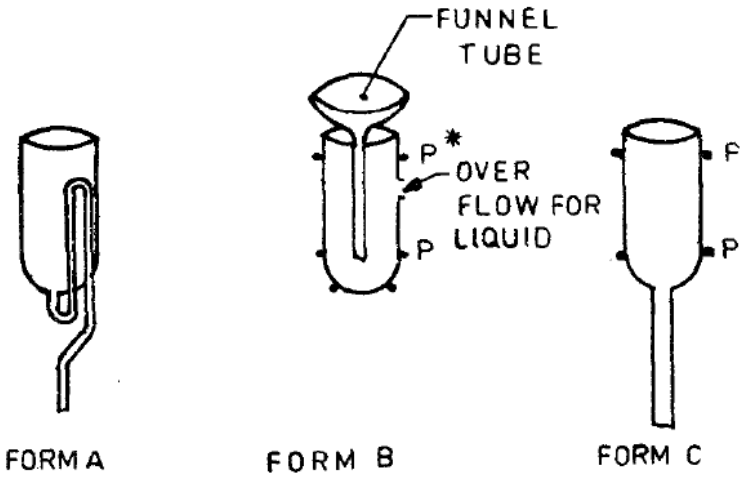


FIG.1 EXTRACTION APPARATUS



*P—Glass points fixed on to furnish passage for vapour round the inner tube

FIG.2 FORMS OF INNER TUBE

furnish a passage for the vapour of the solvent. A little white wool, cotton wool or crushed quartz is placed at the bottom of the tube, as a filtering and regulating medium.

5.2 Conical Flask — 500 ml capacity.

5.3 A Gooch Crucible — provided with filter paper or asbestos.

5.4 Drying Oven — capable of heating up to 110 ± 2°C.

5.5 Beaker — 250 ml capacity.

5.6 Water Bath — capacity of heating up to 70 to 80°C.

5.7 Burrette — graduated to 0.1 ml.

5.8 Graduated Cylinder — 100 ml capacity.

5.9 Titration FIask — 500 ml capacity.

5.10 Steam Oven — for drying.

5.11 Thermometer — capable of measuring a temperature up to 120°C.

5.12 Porcelain Beaker — 250 ml capacity.

5.13 Burner

6 REAGENTS

6.0 Quality of Reagents — Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests and distilled water (*see* IS 1070) shall be used where the use of water as reagent is intended.

6.1 Commercial Pyridine

6.2 Alcohol — 50 percent (*v/v*).

6.3 Sodium Hydroxide Solution — 2 percent (*w/v*).

6.4 Dilute Hydrochloric Acid — 1 percent (*v/v*).

6.5 Ether (Diethyl ether)

6.6 Pure Concentrated Sulphuric Acid (AR Grade)

6.7 Potassium Permanganate Solution — N/50.

6.8 Solvent Mixture — A mixture of 75 parts of cresol (> 98 percent purity) with 25 parts of heavy petroleum spirit (b p 155° to 170°C).

6.9 Sulphuric Acid — 80 percent (*w/v*).

6.10 Dilute Ammonia — 10 percent (*w/v*).

7 PROCEDURE

7.1 Estimation of Indigo on Textiles

7.1.1 Weigh the required quantity of the dyed textile (*see* **3.1**) accurately after heating it in an air oven at 105 ± 3°C for 2-4 hours to constant weight and pack it loosely into the inner tube of the soxhlet apparatus (*see* Fig. 1).

7.1.2 Take 100 ml of commercial pyridine in the boiling flask of the soxhlet apparatus and heat it over wire-gauze or upon an air bath. Continue the extraction until the extract no longer has a blue colour. This usually requires two hours. The thickest material can be completely extracted in four hours at most.

NOTE — Instead of pyridine, a cresol mixture (see 6.8) may also be used for extraction.

7.1.3 Distil down the extract to about 20 or 30 ml and cool the extraction flask along with contents when the greater part of the indigo separates in well-formed bronzy crystals. To complete the precipitation add to it 100 ml of 50 percent alcohol.

7.1.4 Heat the contents of the flask to boil for about 10 to 20 minutes and filter through a Gooch crucible provided with filter paper or asbestos into a beaker.

7.1.5 Wash the precipitate on filter successively with hot (50-60°C) 50 percent alcohol, hot 2 percent sodium hydroxide solution, hot dilute hydrochloric acid solution (1 percent), hot water, alcohol and finally with alcohol and ether.

NOTE — The appearance of the indigo is a guide to its purity. It should form a bronzy crystalline powder testing 100 percent by the tetrasulphonic acid method (*see* IS 11636). A dull appearance shows the presence of impurities.

7.1.6 Collect the washed precipitate on the gooch crucible, the bottom of which is covered with a **7.1.8** Repeat the procedure as given in **7.1.7** for remaining 400 ml solution 4 times and find the average of volume of potassium permanganate used.

7.1.9 Calculate the percentage strength of indigo on the textile by the following formula:

$$S = \frac{0.00147 \times V \times 5 \times 100}{M}$$

where

S = percentage strength of indigo on the textile,

V = average volume of N/50 $KMnO_4$ used (*see* **7.1.8**), and

M = mass of the test specimen (oven dry basis) (*see* **7.1.1**).

little asbestos and dry it for 10 to 20 minutes. Place the crucible in a small beaker containing 15-20 ml of pure concentrated sulphuric acid and heat in water bath at 70 to 80°C for about 45 minutes.

7.1.7 Take the solution in the titrating flask of 500 ml capacity and then make it up to the mark with distilled water. Titrate 100 ml of this solution in 200 ml water against N/50 potassium permanganate solution till end point is obtained (see Note). Note the volume of N/50 potassium permanganate solution required to reach the end point.

NOTE — The end point is obtained when the solution has a pale yellow or orange colour free from any bluish or greenish tint.

8 REPORT

8.1 Report the percentage strength of indigo (*see* **7.1.9)** present on the textile.

9 SAMPLING

9.1 Lot — The quantity of one definite type and quality of textile material dyed essentially under similar conditions delivered to a buyer against one dispatch note shall constitute a lot.

9.2 Sample shall be drawn so as to be representative of the lot. Sample drawn in accordance with the material specifications or as agreed to between the buyer and the seller shall be held to be representative of the lot.

ANNEX A
(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee,
TXD 07

Organization	*Representative(s)*
Department for Jute and Fibre Technology Institute of Jute Technology, University of Calcutta, Kolkata	PROF A K SAMANTA (***Chairman***)
Ahmedabad Textile Industry's Research Association,	SHRIMATI DEEPALI PLAWAT SHRIMATI FAHIMUNNISA KHATIB (*Alternate*)
Ama Herbals Laboratories Pvt Ltd, Lucknow	SHRI Y A SHAH
Archroma India Pvt Limited, Mumbai	SHRI RAJESH RAMAMURTHY SHRI ASHIM GHOSH (*Alternate*)
Atul Limited (Colors Business), Valsad	SHRI V R SAI GANESH SHRI ARINDAM CHAKRABORTY (*Alternate*)
Bio Dyes India Pvt Ltd, Goa	DR BOSCO HENRIQUES
Central Coir Research Institute, Alappuzha	SHRIMATI ANITA JACOB SHRIMATI SUMI SABESTIAN (*Alternate*)
ICAR – Central Institute for Research on Cotton Technology, Mumbai	DR SUJATA SAXENA DR A S M RAJA (*Alternate*)
Department for Jute and Fibre Technology Institute of Jute Technology, University of Calcutta, Kolkata	DR D DAS
Global Organic Textile Standard, (GOTS), Thane	SHRI RAHUL BHAJEKAR MS PRACHI GUPTA (*Alternate*)
Indian Jute Industries Research Association, Kolkata	DR S K CHAKRABARTI SHRI SANDIP BASU (*Alternate*)
Northern India Textile Research Association, Ghaziabad	DR M S PARMAR
Office of the Textile Commissioner, Mumbai	SHRI GAURAV GUPTA SHRI SANJAY CHARAK (*Alternate*)
SGS India Pvt Ltd, Mumbai	SHRI KARTHIKEYAN K SHRI GAURAV SARASWAT (*Alternate*)
Shree Pushkar Chemicals & Fertilizers Ltd, Mumbai	DR N N MAHAPATRA
Textiles Committee, Mumbai	SHRI KARTIKEYA DHANDA SHRIMATI SHILPI CHAUHAN (*Alternate*)

Organization	*Representative(s)*
The Arvind Mills Limited, Ahmedabad	SHRI RAJARSHI GHOSH SHRI UMASANKAR MAHAPATRA (*Alternate*)
The Bombay Textile Research Association, Mumbai	DR PADMA S VANKAR SHRI M P SATHIANARAYANAN (*Alternate*)
The South India Textile Research Association, Coimbatore	DR PRAKASH VASUDEVAN SHRI S SIVAKUMAR (*Alternate*)
The Synthetic and Art Silk Mills Research Association, Mumbai	SHRIMATI (DR) MANISHA MATHUR SHRIMATI ASHWINI SUDAM (*Alternate*)
U P Textile Technology Institute, Kanpur	DR ARUN PATRA
Wool Research Association, Thane	SHRIMATI SMITA BAIT SHRIMATI (DR) MRINAL CHOUDHARI (*Alternate*)
BIS Directorate General	SHRI J K GUPTA, Scientist E and Head (Textiles) [Representing Director General (*Ex-officio*)]

Member Secretary
SHRI HIMANSHU SHUKLA
Scientist B (Textiles), BIS

Bureau of Indian Standards

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Review of Indian Standards

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: TXD 07(17974).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central:	601/A Konnectus Tower-I, 6th Floor DMRC Building, Bhavbhuti Marg, New Delhi 110 002	2323 7617
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Northern:	Plot No. 4A, Sector 27-B, Madhya Marg Chandigarh 160 019	265 0206, 265 0290
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Publication Unit, BIS, New Delhi, India